

Claims:

1. Method for the automatic determination of selected physical, technical method and colloidal chemistry parameters (for example, the grain size, the distribution of grain sizes, the speed distribution, the particle flux, the hindrance function and indices of structural stability) by means of the determination of the attenuation of radiated waves during the segregation of monodisperse or polydisperse dispersion samples subjected to gravitation or centrifugation, characterised by the following features:

1.1. during the segregation by means of centrifugation or gravitation, the momentary transmission $I_T(t, r)$ characterising the current segregation status of the waves radiated with the intensity $I_0(t, r)$ and/or the instantaneous scattering $I_S(t, r)$ as a function of the position within the samples is repeatedly determined and recorded at high resolution at any arbitrary time for one or more wavelengths over the entire length of the sample or in selected partial sections of it, simultaneously for multiple and even concentrated samples with known and/or unknown physical and colloidal chemistry properties.

1.2. the extinction profile $E_T(t, r)$ is calculated by finding the log of the ratio of $I_0(t, r) / I_T(t, r)$ for the determination of the particle or droplet concentration for the tested dispersion samples as a function of sample position and time.

1.3. from these extinction profiles $E_T(t, r)$ determined at different times ($t_1 \dots t_n$) and the local adjustment made in these time segments ($t_{(n-1)} - t_{(n)}$), segregation speeds are calculated for any constant extinction values.

1.4. from the ratio of the segregation speeds determined for specific extinction percentiles, a polydispersity index is calculated, which is characteristic for the polydispersity of the density or the particle or droplet size.

1.5. extinction-weighted distributions of the grain size are calculated from extinction profiles $E_T(t, r)$ for selectable times according to Equation A while standardising on the maximum extinction for this profile.

1.6. the local and temporal change of the particle or droplet concentration can be determined by taking into account the substance-specific extinction concentration relationship.

1.7. the substance-specific extinction–concentration relationship is calculated through the simultaneous segregation of samples of the substance system to be measured with known, varying volume concentrations, whereby the concentration effect on the extinction is calculated while taking into account the repeated scattering, for example according to Equation B

and/or

- 1.8. the volume-weighted distributions of the grain size are calculated according to Equations A and C from any extinction profiles acquired at time t according to 1.2, whereby

1.8.1. the volume-specific extinction cross section that is dependent on particle size and that is required for doing so is calculated according to Mie while including the device constants from the known optical substance parameters, or

1.8.2. as an alternative to 1.8.1, the method allows the experimental determination of the volume-specific extinction cross section that is dependent on particle size if the extinction of at least two monodisperse reference samples is determined corresponding to 1.2, or

1.8.3. as an alternative to 1.8.1, the method allows the experimental determination of the volume-specific extinction cross section that is dependent on particle size if the course of the extinction is determined during the segregation of at least one polydisperse substance system with similar optical characteristics corresponding to 1.2

and/or

- 1.9. using the volume-weighted distribution of particle sizes determined in 1.8, the particle size dependency for the volume-specific extinction cross section determined in 1.8.1 – 1.8.3, and the concentration-dependent extinction determined in item 1.6, each radial position and the particle size associated with it via Equation A is assigned a volume concentration corresponding to Equation D

and/or

- 1.10. the flux density function standardised to the centrifugation constant (Equation F) is determined from the change in the concentration of the samples with known starting concentration

and/or

- 1.11. the concentration-dependent hindrance function for the substance system is determined corresponding to Equations E, E^* and F

and/or

1.12. the volume-weighted distribution of the Stokes equivalent diameter for the case of hindrance functions not equal to 1 is determined by iteratively repeating Equation G instead of Equation A for the steps described in 1.2 through 1.11 until the difference between the concentration profiles of consecutive steps are less than a value to be provided in advance, or if the allowance for the hydrodynamic impediment (Equation E) is provided by means of another suitable mathematical algorithm, for example via the definition of a cost function.

2. Method according to claim 1, characterised in that the determination of the grain sizes and their distribution is possible for dispersed particles with density both greater as well as less than that of the dispersion medium.

3. Method according to claim 1, characterised in that in place of the position-dependent extinction profile $E_T(t, r)$ at time t , the extinction is determined as a function at a freely selectable position or over a range $(r + \delta r)$ of the sample, and the distribution of grain sizes is calculated from it analogously to the above calculation.

4. Method according to claim 1, characterised in that the apparent relative viscosity can be calculated as a function of the concentration by volume from the hindrance function determined according to 1.6, taking into account the concentration by volume.

5. Method according to claim 1, characterised in that the sedimentation type and the critical concentration by volume for the use of consolidation phenomena can be determined from the change in the segregation speed during the segregation.

6. Method according to claim 1, characterised in that the ascertainable range of the distribution of sizes as well as the resolution with respect to the distribution of grain sizes can be increased by varying the number of revolutions and the measurement time intervals.

7. Method according to claim 1 and 3, characterised in that the mass density distribution of the sample is calculated from the extinction profile $E_T(t, r)$ for a known distribution of grain sizes.

8. Method according to claim 1 and 3, characterised in that for mixtures of substances of different densities, the distribution of grain sizes for the individual substance components is calculated from the extinction profiles for the segregation of dispersions with different densities for the dispersion medium.

9. Method according to claim 1, 3 and 4, characterised in that indices for the consolidation behavior of the dispersion samples can be computed from the sediment levels for gradually changed revolutions related to the respective operative centrifugal force.

10. Method according to claim 1 through 9, characterised in that the control of the segregation analyser and the measurement sensor, including radiation source, sample management and data transfer, data handling and data storage, as well as all steps of analysis and the documentation of the results, takes place by means of software supported by a database.

11. Device for the automatic determination of selected physical, technical method and colloidal chemistry parameters (for example, the grain size, the distribution of grain sizes, the speed distribution, the particle flux, the hindrance function and indices of structural stability), consisting of a PC-controlled multi-sample receptacle unit arranged vertically or horizontally with a spectrometric measurement device with a source producing monochromatic parallel radiation, which registers, digitises and stores the radiation intensity scattered or transmitted by the respective dispersion sample over the entire length of the sample simultaneously or shifted temporally during the segregation, resolved for location and time.

12. Device according to 11, characterised in that different cuvettes matched to the measurement task and/or the dispersion sample with respect to the optical path length and the materials can be used, the cuvette type is detected automatically, and the parameters required for the analysis of the measurement results are automatically made available via database entries for the calculation of the parameters to be analysed.

13. Device according to 11 and 12, characterised in that radiation sources of different monochromatic wavelengths, whose radiation intensity $I_0(t, r)$ can be varied, are also used electively in an alternating fashion, depending on the sample and measurement tasks.

14. Device according to 11 through 13, characterised in that the measurement range can be controlled by thermostat and the measurements can be carried out at selectable temperatures both under as well as over room temperature.

15. Device according to 11 through 14, characterised in that the multi-sample receptacle unit is designed as a rotor, and is driven by a motor with programmable variable and/or constant revolutions.

16. Device according to 11 through 14, which as an alternative to 15 has a multi-sample receptacle unit, which makes possible the acceptance of samples placed vertically for segregation in the gravitational field.